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# Phase behavior in mixtures of cationic and anionic surfactants in aqueous solutions

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**Abstract** Phase behavior of cationic/anionic surfactant mixtures of the same chain length ($n = 10, 12$ or 14) strongly depends on the molar ratio and actual concentration of the surfactants. Precipitation of catanionic surfactant and mixed micelles formation are observed over the concentration range investigated. Coacervate and liquid crystals are found to coexist in the transition region from crystalline catanionic surfactant to mixed micelles.

The addition of oppositely charged surfactant diminishes the surface charge density at the mixed micelle/solution interface and enhances the apparent degree of counterion dissociation from mixed micelles. Cationic surfactants have a greater tendency to be incorporated in mixed micelles than anionic ones.

**Key words** Cationic/anionic surfactant mixtures – phase behavior – catanionic surfactant precipitation – mixed micelles – counterion binding

## Introduction

Aqueous systems containing mixtures of oppositely charged surfactants have recently attracted the attention of numerous investigators [1–7]. Strong synergistic effects are observed for both their bulk and surface properties. General observations include: separation into a dilute solution and a solid precipitate in equimolar mixtures, or the formation of mixed micelles in an excess of one of the components. Monomer-precipitate equilibria seen to be dominant in equimolar mixtures while nonequimolar mixtures exhibit a phase transition from precipitate to mixed micelles [6]. We have recently described the structure and composition of the solid alkylammonium alkyl sulfate formed from equimolar mixtures [7]. In this work the results of systematic investigations of the phase behavior of mixtures with varying molar ratios of reactant components (cationic and anionic surfactant with 10, 12 and 14 C-atoms in the chain) are presented. It is shown that the

ionic nature of the polar head groups of the surface active substance and its hydrocarbon chain length significantly influence the phase behavior of cationic/anionic surfactant aqueous solution.

## Experimental

### Materials

Decylammonium chloride (DeACl), dodecylammonium chloride (DDACl) and tetradecylammonium chloride (TDACl) were prepared by neutralization of alkylamine with HCl [8]. Commercially obtained sodium decyl sulfate (SDeS), sodium dodecyl sulfate (SDS), and sodium tetradecyl sulfate (STDS) were purified by repeated recrystallization. The surfactant purity was checked by observing that the surface tension ($\gamma$) (Interfacial Tensiometer K8, Krüss, Hamburg) vs. concentration ($c$) curve has no minimum. The critical micelle concentration

(CMC) determined for each surfactant (surface tension or conductivity measurements (Conductometer, Radiometer)) were in good agreement with the literature data [9]. Systems were prepared either with a constant anionic and increasing cationic component or vice versa. The experiments were carried out at 298 K and 303 K.

Methods and interpretation of data

The structure and composition of solid phases were characterized by polarizing microscopy, x-ray diffraction, infrared spectroscopy, and elemental analysis [7].

The precipitation/solubility boundary was taken as the maximum concentration of reactant which did not result in the solid phase formation as observed visually [10]. Optical microscopy (Leitz, Orthoplan) was used to detect small amounts of crystals in the systems. The monomer-precipitate equilibrium
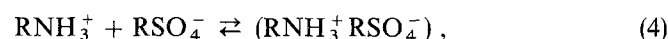
$$RNH_3^+ + RSO_4^- \rightleftarrows RNH_3RSO_4 \tag{1}$$

is described by the solubility product $(K_{sp}^0)$

$$K_{sp}^0 = c(RNH_3^+)c(RSO_4^-)f^2 , \tag{2}$$

where $R$ denotes $C_nH_{2n+1}$, $RNH_3^+$ is the alkylammonium cation, $RSO_4^-$ is the alkyl sulfate anion, $RNH_3 RSO_4$ is the alkylammonium alkyl sulfate salt (catanionic surfactant), $c$ is the equilibrium concentration, and $f$ the mean activity coefficient. For univalent ions $f$ is given by the Debye–Hückel limiting law

$$-\log f = AI^{1/2}/(1 + I^{1/2}) , \tag{3}$$

where $I$ is the ionic strength expressed in $mol\, dm^{-3}$ and $A$ is a temperature-dependent constant. At high ionic strength $(1\, mol\, dm^{-3}\, NaCl)$ $f$ cannot be calculated by means of Eq. (3). The value $f = 0.657$ was taken from the literature [11]. Formation of ion pairs can be described by

$$RNH_3^+ + RSO_4^- \rightleftarrows (RNH_3^+RSO_4^-) , \tag{4}$$

and the corresponding association constant $(K_a^0)$ is

$$K_a^0 = c(RNH_3^+RSO_4^-)/f^2c(RNH_3^+)c(RSO_4^-) , \tag{5}$$

where $(RNH_3^+RSO_4^-)$ represents the soluble ion pair. The liquid phase was analyzed by specific conductivity and potentiometric (Ion Analyzer, Orion Research) measurements [5, 6].

The CMC for mixed surfactant systems was determined from the plots of the specific conductance $(x)$ vs. concentration $(c)$. The apparent degree of dissociation $(a)$ was calculated from conductivity and potentiometric measurements. To a first approximation $\alpha$ was taken as the ratio of the slope of the $x$ vs. $c$ lines above and below the CMC [12]. Although $\alpha$-values calculated on the basis of

conductivity data do not strictly represent the degree of counterion dissociation, since $x$ depends on the micellar size, electroviscosity and other ions present, it satisfactorily reflects the change in the micellar surface charge density.

The activities of $Na^+$ and $Cl^-$ ions were determined with the corresponding ion-selective electrodes [6]. The $\alpha$-value was determined as the ratio of the slopes above and below breaks in curves of the concentration of free counterions (counterions not bound to the micelle) $(c_f)$ vs. the concentration of surfactant added $(c_t)$ [13].

The fraction of counterions bound to micelles $(F)$ was calculated by

$$F = (c_t - c_f)/c_t , \tag{6}$$

and represents the micelle counterion binding capacity.

## Results and discussion

Figure 1 shows the precipitation/solubility boundaries and the decrease of the CMC of surfactant in excess for mixtures of cationic/anionic surfactant of the same chain length $(n = 10$, Fig. 1a; $n = 12$, Fig. 1b; $n = 14$, Fig. 1c). Precipitation/solubility boundaries exhibit a linear part near the equimolar region and deviation from linearity with increasing concentration of either cationic or anionic component. With increasing alkyl chain length the precipitation/solubility boundary is shifted toward lower surfactant concentration.

The interpretation of the precipitation/solubility boundary at low ionic strength is given by numerical procedure using results obtained in the absence and the presence of neutral electrolyte [5]. Plots of $\log a_{RNH_3^+}$ vs. $\log a_{RSO_4^-}$ obtained for high and low ionic strength (only the linear part observed near the equimolar region) are shown in Fig. 2.

At high ionic strength the ion-pairing can be neglected $(K_a^0 = 0)$. Equilibrium dissolution constants can be calculated from each experimental point. The effect of surfactant alkyl chain length $(n)$ on $K_{sp}^0$ is shown in Fig. 3. The decrease in the $K_{sp}^0$-value with increasing number of $CH_2$-groups in the hydrocarbon chain can be described by the relation

$$\log K_{sp}^0 = -0.51\, n - 4.65 \tag{7}$$

The calculated value of the $K_{sp}^0$ at low ionic strength based on the $K_{sp}^0$ value determined in the presence of NaCl exhibits a lower solubility than is obtained from experiments. Therefore, the calculation of $K_{sp}^0$ at low ionic strength requires consideration of association reaction with appropriate association constants [5]. If the concen-
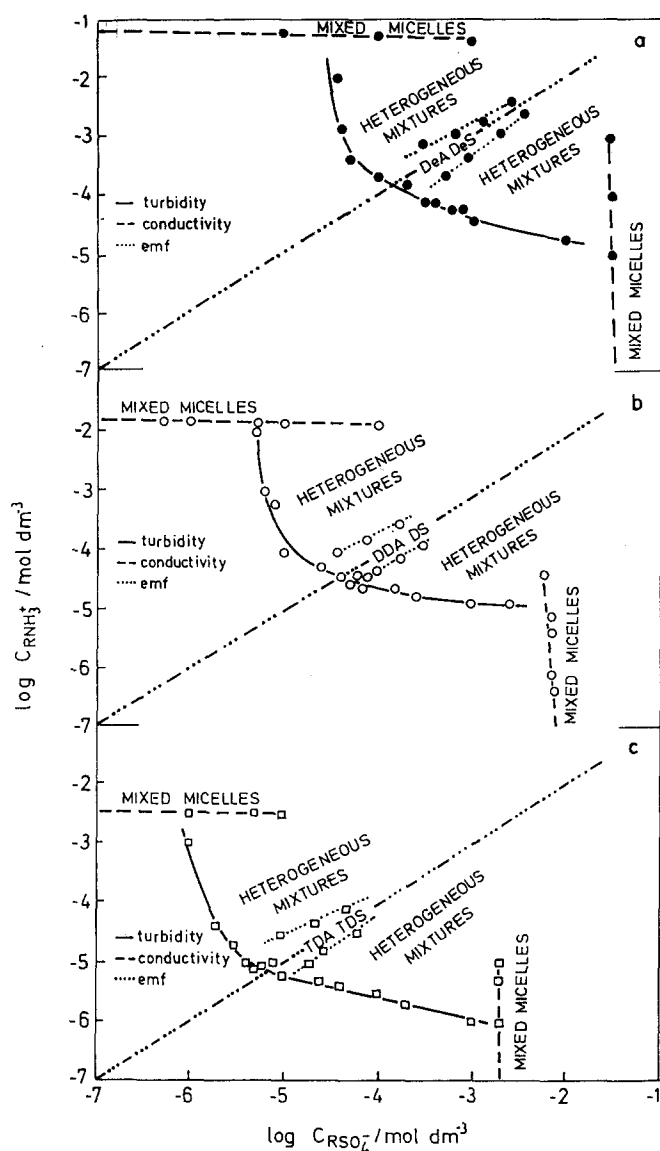
Fig. 1 Phase behavior of alkylammonium chloride/alkyl sulfate systems. The full lines correspond to precipitation/solubility boundaries; dashed lines correspond to the change of the CMC of the surfactant in excess. Dotted lines denote the range of pure crystalline $RNH_3RSO_4$ formation. Equimolar lines are denoted as — · · — · · · —. a) DeACl/SDeS mixture; b) DDACl/SDS mixture (after ref. [6]); c) TDACl/STDS mixture. Temperature is 298 K

trations determined from the linear part of solubility/precipitation boundary in the equimolar region are taken as the ion pair concentrations, by combining Eqs. (1) and (5), values of log $K_a^0 = 5.65$, 6.00 and 6.25 are calculated for decyl, dodecyl, and tetradecyl chains, respectively. It seems that ion-pairing takes place only in a narrow range of concentrations close to the equimolar region. Such a conclusion is in accordance with the previous results, sugges-

ting that a soluble ion pair may be formed in cationic/anionic mixtures before precipitation [5, 14].

Deviation from the calculated data (dotted lines in Fig. 2b) in excess of either $RNH_3^+$ or $RSO_4^-$ indicates further association. According to Stellner et al., deviation from linearity in the precipitation/solubility boundary corresponds to the points where micelles begin to form in solution [1].

Close to the equimolar range only solid crystalline $RNH_3RSO_4$ is formed and emf measurements indicated no binding of counterions. IR analysis indicated electrostatic interactions between cationic and anionic headgroups; x-ray data indicated lamellar structures for the precipitates formed [7]. Krafft points for catanionic surfactant as determined from specific conductivity measurements are 35 °C, 45 °C and 54.5 °C for decyl, dodecyl and tetradecyl hydrocarbon chains, respectively [7].

When the overall bulk composition is shifted from equimolar, the coexistence of several phases is observed (Fig. 1, heterogeneous mixture region). Examination of the systems by polarizing microscope showed that the compositions of the heterogeneous microstructures vary continuously with concentration. As the overall surfactant concentration rises coacervate and liquid crystals appear; crystalline cationic surfactant is always present.

Similar observations have been reported earlier for aqueous mixtures of two anionic surfactants: one a single-tail (SDS) and the other a double-tail alkylarylsulfonate (sodium 4-(1′heptynonyl)benzenesulfonate (SBS)) (15). Experimental conditions above the Krafft point of SDS and below the Krafft point of SBS, enable SDS to exist as monomer and micelles and SBS to exist as monomer and crystalline phase. It is shown that SDS micelles can solubilize SBS crystals to form mixed micelles and that SBS crystals can incorporate SDS to form mixed crystals.

In this work the temperature was above the Krafft points of the individual cationic and anionic surfactants, but below the Krafft point of the catanionic surfactant complex. We could not determine whether the phase in the transition region with catanionic surfactant is composed of only one surfactant or their mixture. However, it seems that the formation of catanionic surfactant shifts the solution composition in favor of the surfactant in excess [5]. More information about the transition from precipitate to coacervate, liquid crystals, and mixed micelles can be obtained on the basis of kinetic precipitation experiments. [7, 16]. That question is the subject of current investigations.

Close to the CMC of the surfactant in excess the turbid heterogeneous mixture becomes clear due to the formation of mixed micelles. Addition of small amounts of cationic surfactant to the anionic surfactant, near or above its CMC, and vice versa, results in a shift of the CMC of the surfactant in excess toward lower concentration. Two

**Fig. 2** a) Plots of alkylam-monium ion (RNH$_3^+$) activity vs. alkylsulfate ion (RSO$_4^-$) activity change for different chain length. Open symbols represent data obtained at low ionic strength, while filled symbols correspond to data obtained at 1 mol dm$^{-3}$ NaCl. b) Small diagram shows precipitation/solubility boundaries at low ionic strength. Dotted lines are calculated by taking equilibrium constant for ion pair association. Temperature is indicated
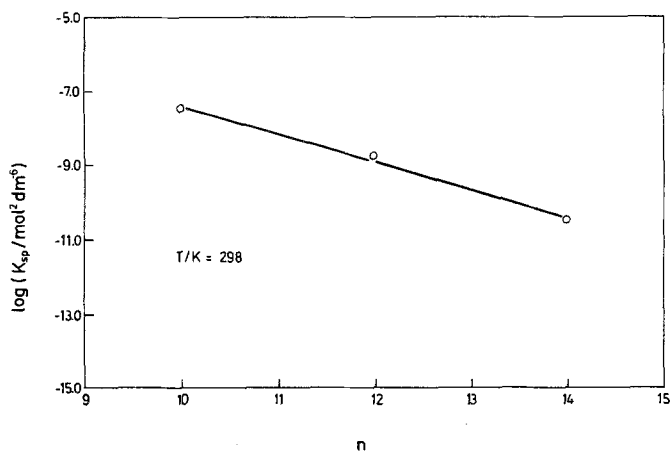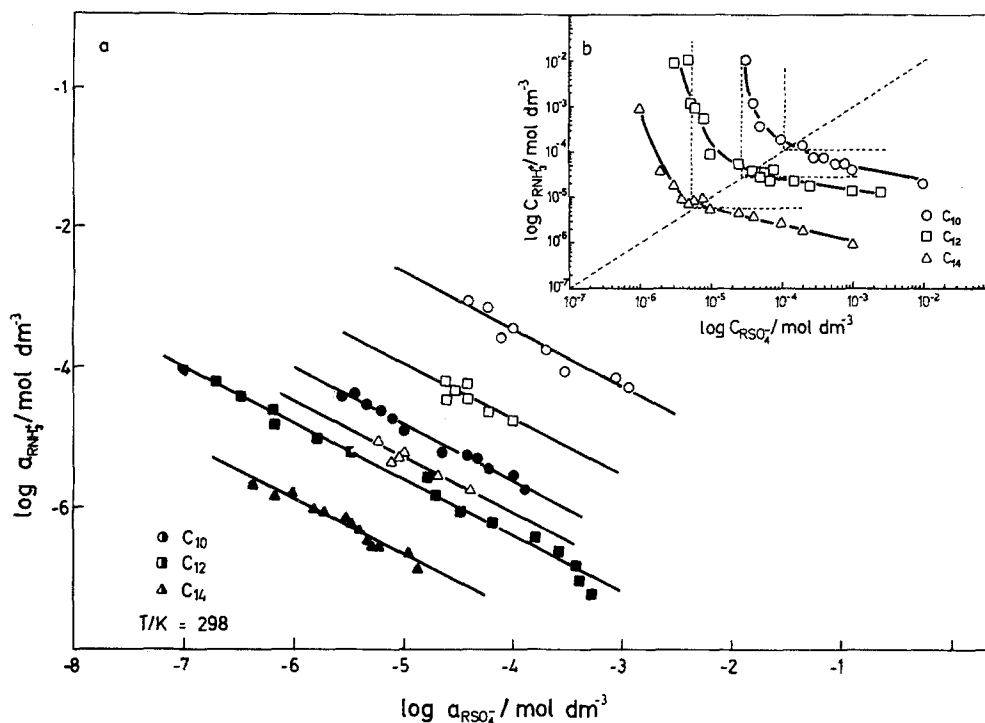


**Fig. 3** Solubility product ($K_{sp}$/mol$^2$ dm$^{-6}$) of RNH$_3$RSO$_4$ precipitate as a function of alkyl chain length ($n$) of RNH$_3^+$ and RSO$_4^-$, respectively. Temperature is indicated
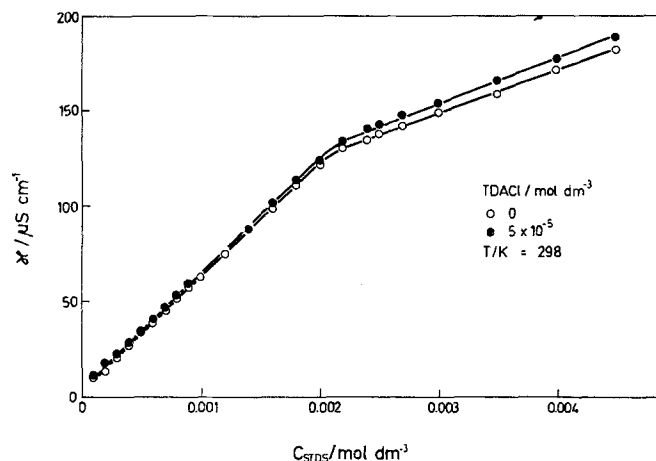
**Fig. 4** Specific conductivity ($\varkappa$) as a function of STDS concentration in TDACl/STDS mixtures. Constant TDACl concentration and temperature are indicated

main factors responsible for the lowering of the CMC are [17]; i) an increase in the entropy of mixing of the surfactant with opposite charge and ii) a decrease in the electrical work of micellization due to the decrease of the surface charge density caused by solubilized surfactant of opposite charge.

Electrical properties of mixed micelles rich in one of the surfactants were investigated by specific conductivity and potentiometric measurements. Figure 4 shows the typical

conductivity curves for investigated systems. It can be seen that the intersection point is shifted toward lower surfactant concentration and the slope above the CMC increases with the addition of small amounts of oppositely charged surfactant. Obtained CMC values are given in Fig. 1 and α-values are listed in Table 1.

Potentiometric measurements revealed that the mixed micelle/solution interface includes the headgroups of both surfactants and counterions of the surfactant in excess

**Table 1.** Apparent degree of counterion dissociation from mixed micelles ($\alpha$) calculated from conductivity measurements for a) cationic/anionic mixtures with constant $RNH_3Cl$ and increasing $RSO_4Na$ concentration and b) constant $RSO_4Na$ and increasing $RNH_3Cl$ concentration, for reactants with different chain length ($n$)

| $n$ | 10 | 12* | 14 | 14 |
|---|---|---|---|---|
| | | 298 K | | 303 K |
| a) $C_{RNH_3Cl}$/mol dm$^{-3}$ | | $\alpha_{Na}$ | | |
| 0 | 0.47 | 0.37 | 0.36 | 0.39 |
| $10^{-6}$ | – | 0.61 | 0.39 | 0.40 |
| $5 \times 10^{-6}$ | – | 0.61 | 0.40 | 0.42 |
| $10^{-5}$ | 0.47 | 0.62 | 0.41 | 0.42 |
| $5 \times 10^{-5}$ | – | 0.74 | 0.44 | 0.42 |
| $10^{-4}$ | 0.50 | 0.71 | 0.47 | 0.56 |
| $10^{-3}$ | 0.58 | – | – | – |
| b) $C_{RSO_4Na}$/mol dm$^{-3}$ | | $\alpha_{Cl}$ | | |
| 0 | 0.36 | 0.28 | 0.27 | 0.31 |
| $10^{-6}$ | – | 0.32 | – | 0.34 |
| $5 \times 10^{-6}$ | – | 0.34 | – | 0.38 |
| $10^{-5}$ | 0.40 | 0.41 | – | 0.37 |
| $5 \times 10^{-5}$ | – | 0.31 | – | 0.36 |
| $10^{-4}$ | 0.44 | 0.33 | – | 0.35 |

*corresponds to the results taken from ref. [6]

[18]. $\alpha$-values obtained in excess of $RNH_3Cl$ correspond approximately to the apparent degree of dissociation for chloride ions, $\alpha_{Cl}$, while in excess $RSO_4Na$ it corresponds to the apparent degree of dissociation from mixed micelles for sodium ions, $\alpha_{Na}$. At 298 K $\alpha_{Cl}$ could not be determined for the mixed micelles formed in the systems with excess of tetradecylammonium chloride since crystallization began immediately after the CMC was reached. At 303 K crystallization is avoided and a second slope in the $x$ vs. $c$ curve could be determined. It can be seen that temperature only slightly affects counterion binding to mixed micelles (Table 1). In a single surfactant system the $\alpha$-value decreases with increasing number of $CH_2$-groups. Increased counterion dissociation with the addition of oppositely charged surfactant (Table 1) is, in general, a consequence of the decrease in the surface charge density. It can be readily seen that the trend of increasing values of $\alpha$ as a function of the concentration of oppositely charged surfactant differs between sodium and chloride counterions. Mixed micelles formed with the cationic component in excess show smaller increases of $\alpha_{Cl}$ than $\alpha_{Na}$ for mixed micelles formed with the anionic component in excess. Observed differences in $\alpha_{Cl}$ and $\alpha_{Na}$ changes may be correlated to the change in mixed micelle size [19] and the nature of the interaction between headgroups and counterions. It implies that cationic surfactant (especially DDACl) has a greater tendency to be incorporated in anionic micelles than anionic surfactant in cationic micelles.
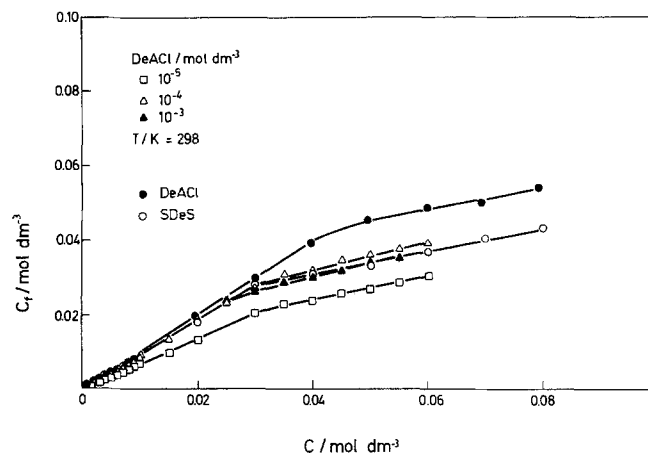


**Fig. 5** Concentration of free sodium ions ($C_f$/mol dm$^{-3}$) vs. concentration of SDeS in DeACl/SDeS mixtures. Constant DeACl concentration and temperature are indicated. Changes of free chloride ions in single DeACl (●) and free sodium ions in single SDeS (○) are given for comparison
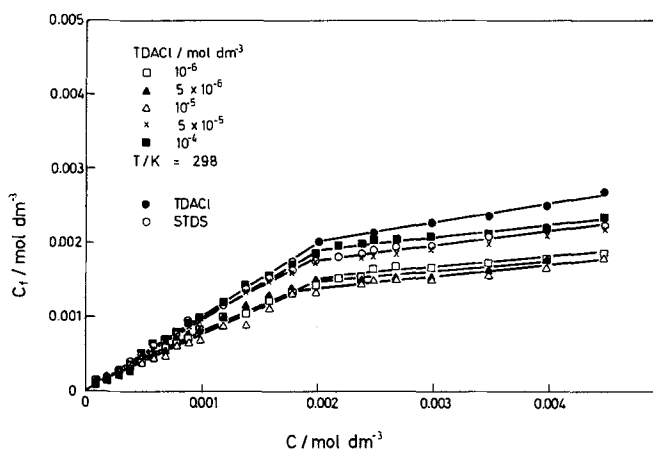


**Fig. 6** Concentration of free sodium ions ($C_f$/mol dm$^{-3}$) vs. concentration of STDS in TDACl/STDS mixtures. Constant TDACl concentration and temperature are indicated. Changes of free chloride ions in single TDACl (●) and free sodium ions in single STDS (○) are given for comparison

Direct proof of counterion binding to the mixed micelles was provided by potentiometric measurements. The results of emf measurements are presented as the change of concentration free sodium ions vs. SDeS (Fig. 5) and STDS (Fig. 6) concentration for the system prepared with constant DeACl and TDACl concentration, respectively. Results for cationic surfactants alone are shown for comparison (concentration of free chloride ion vs. DeACl or TDACl concentration, respectively). Similar behavior is observed for mixtures with $n = 12$ [6]. Below the CMC, the surfactants behave as a strong electrolyte and electrode

**Table 2.** Apparent degree of sodium ion dissociation ($\alpha_{Na}$) from mixed micelles formed in systems with constant $RNH_3Cl$ and varying $RSO_4Na$ and apparent degree of chloride ion dissociation ($\alpha_{Cl}$) from single micelles of $RNH_3Cl$ as determined by potentiometric measurements. Columns "1" correspond to lower $RSO_4Na$ concentration while columns "2" correspond to the micellar region. $n$ is the chain length

| $n$ | 10 | | 12* | | 14 | |
|---|---|---|---|---|---|---|
| $C_{RNH_3Cl}$/mol dm$^{-3}$ | | | $\alpha_{Na}$ | | | |
| | 1 | 2 | 1 | 2 | 1 | 2 |
| 0 | — | 0.33 | — | 0.28 | — | 0.19 |
| $10^{-6}$ | — | — | — | — | 0.72 | 0.21 |
| $5 \times 10^{-6}$ | — | — | — | — | 0.76 | 0.20 |
| $10^{-5}$ | 0.63 | 0.31 | — | — | 0.68 | 0.22 |
| $5 \times 10^{-5}$ | — | — | — | — | 0.69 | 0.27 |
| $10^{-4}$ | 0.93 | 0.43 | 0.98 | 0.63 | 0.68 | 0.23 |
| $5 \times 10^{-4}$ | — | — | 0.96 | 0.74 | — | — |
| $10^{-3}$ | 0.87 | 0.43 | 0.94 | 0.79 | — | — |
| $C_{RSO_4Na}$/mol dm$^{-3}$ | | | $\alpha_{Cl}$ | | | |
| 0 | | 0.26 | | 0.25 | | 0.23 |

*corresponds to the results taken from ref. [6].

response is Nernstian; i.e., $c_f$ is equal to the concentration of surfactant added. At and above the CMC, the $c_f$-value is lower than the concentration of simple surfactant due to the binding to micelles. If cationic surfactant is added to anionic surfactant below the $CMC_{RSO_4Na}$, $c_f^{Na}$ is lower than the concentration of anionic surfactant added due to binding to mixed micelles. Two breaks are observed in $c_f^{Na}$ vs. $c_{RSO_4Na}$ curves; one in the vicinity of the conventional $CMC_{RSO_4Na}$ and the other at a lower $RSO_4Na$ concentration, indicating two regions of mixed micelles formation. Calculated $\alpha_{Na}$-values are listed in Table 2. High $\alpha_{Na}$-values at lower anionic surfactant concentration ($\alpha_{Na}$-values in column 1) indicate the formation of mixed micelles with small excesses of anionic component. The second break in the vicinity of the $CMC_{RSO_4Na}$ corresponds to mixed micelles with a higher content of anionic component. Increase of $\alpha_{Na}$ in the vicinity of the $CMC_{RSO_4Na}$ ($\alpha_{Na}$-values in column 2) with $RNH_3Cl$ concentration increasing indicates a decrease in counterion binding on mixed micelles. The above-described changes are in accordance with the results of specific conductivity measurements.

The fraction of sodium ions ($F$) bound to simple anionic and mixed micelles is illustrated in Figs. 7a and b for mixtures with $n = 10$ and $n = 14$, respectively. $F$ increases with the anionic surfactant concentration. The existence of several discontinuities indicates complex changes in counterion binding to the mixed micelles. When DeACl is added to SDeS (Fig. 7a) binding of sodium ions to mixed micelles starts at a lower SDeS concentration due to mixed micelle formation. Mixtures with the lowest DeACl concentration exhibit a higher binding capacity for sodium ions than single SDeS micelles. This cross-section corresponds to the isotropic region (Fig. 1a). Two other cross-sections extend into the turbid region; the decrease in binding capacity can be explained by the formation of catanionic surfactant complexes, DeADeS.
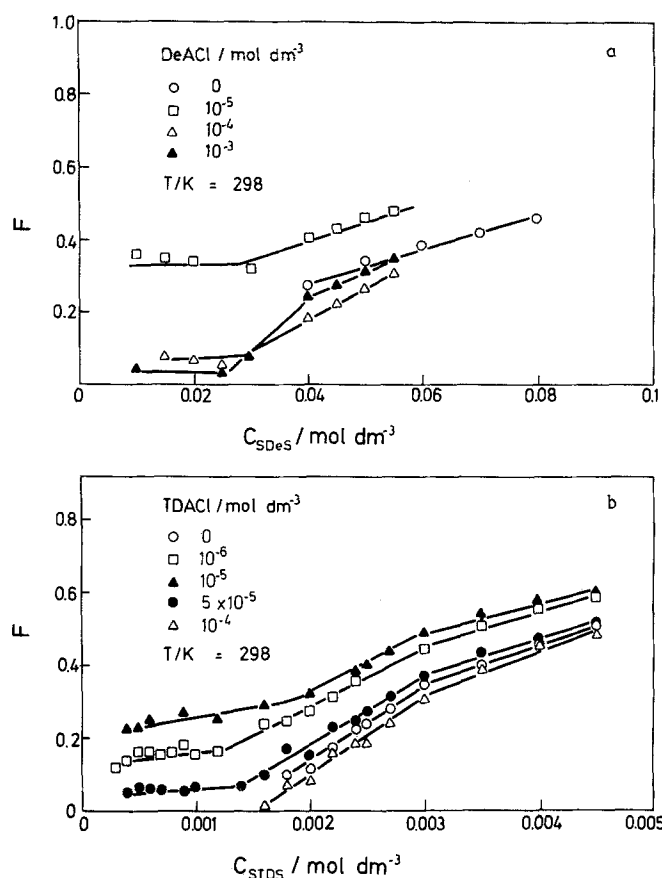


**Fig. 7** Fraction of bound sodium ions ($F$) on micelles vs. SDeS concentration in DeACl/SDeS mixtures (a) and vs. STDS concentration in TDACl/STDS mixtures (b). Constant concentration of cationic surfactant and temperature are indicated

The fraction of sodium ion bound to a single STDS micelle shows a break after the $CMC_{STDS}$ (Fig. 7b, at 0.003 mol dm$^{-3}$). Quite generally, a second break after the conventional CMC is usually called the second CMC. It may be attributed mainly to a change in micelle size and

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shape [20–23]. If TDACl is added, the binding of sodium ions begins at lower STDS concentrations due to a mixed micelles formation. Binding capacity changes are similar to those observed in the mixtures of SDeS/DeACl.

The presence of oppositely charged surfactant in mixed micelles reduces electrostatic repulsion between ammonium and sulfate headgroups at the micelle/solution interface. We believe that both the reduction of electrostatic repulsion at the mixed micelle/solution interface due to the presence of cationic and anionic headgroups and a change of mixed micelle geometry enhance counter ion dissociation. As pointed out by Scheuing and Weers [4], changes in the packing of methylene tails in the mixed micelle interior as well as the surfactant headgroups at the mixed micelle/solution interface may contribute to the changes in micelle ionization. At present, information about sizes and shapes in the systems examined in unavailable but should be obtained in future research.

## Conclusions

The phase behavior of cationic/anionic surfactant mixtures strongly depends on the molar ratio and the concentration of surfactants present. An increase in the alkyl chain length increases the tendency for both precipitation and micellization to occur.

From equimolar mixtures a catanionic surfactant complex precipitates; before the precipitation ion-pairs exist in solution. The increase of hydrocarbon chain length decreases the $K_{sp}^0$ of catanionic surfactant.

When the overall bulk composition is shifted from the equimolar region, crystalline catanionic surfactant complex, coacervate, and liquid crystals coexist (transition region from monomer-solid phase to monomer-mixed micelle equilibria). Close to the CMC of the surfactant in excess, the turbid heterogeneous mixtures become clear due to mixed micelle formation. Interactions between oppositely charged headgroups diminish the charge density at the mixed micelle/solution interface and the $\alpha$-value is enhanced. The charge on mixed micelles varies with bulk composition. Cationic surfactant has a greater tendency to be incorporated into anionic micelles than anionic surfactants into cationic micelles.

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